

FORM PTO-1290 (Modified) REV 10-93		U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE		ATTORNEY'S DOCKET NUMBER ATOCM 151	
TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371				U.S. APPLICATION NO. (IF KNOWN, SEE 37 CFR 09/341543	
INTERNATIONAL APPLICATION NO. <u>PCT/FR98/02409</u>		INTERNATIONAL FILING DATE 12 November 1998		PRIORITY DATE CLAIMED 14 November 1997	
TITLE OF INVENTION <b>PRESSURE-SENSITIVE ADHESIVE WHICH CAN BE APPLIED WHILE HOT</b>					
APPLICANT(S) FOR DO/EO/US <b>RADIGON, Eric et al.</b>					
Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:					
<ol style="list-style-type: none"> <li><input checked="" type="checkbox"/> This is a <b>FIRST</b> submission of items concerning a filing under 35 U.S.C. 371.</li> <li><input type="checkbox"/> This is a <b>SECOND</b> or <b>SUBSEQUENT</b> submission of items concerning a filing under 35 U.S.C. 371.</li> <li><input type="checkbox"/> This is an express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(1).</li> <li><input type="checkbox"/> A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.</li> <li><input checked="" type="checkbox"/> A copy of the International Application as filed (35 U.S.C. 371 (e) (2))           <ol style="list-style-type: none"> <li><input type="checkbox"/> is transmitted herewith (required only if not transmitted by the International Bureau).</li> <li><input checked="" type="checkbox"/> has been transmitted by the International Bureau.</li> <li><input type="checkbox"/> is not required, as the application was filed in the United States Receiving Office (RO/US).</li> </ol> </li> <li><input checked="" type="checkbox"/> A translation of the International Application into English (35 U.S.C. 371(c)(2)).</li> <li><input checked="" type="checkbox"/> A copy of the International Search Report (PCT/ISA/210).</li> <li><input checked="" type="checkbox"/> Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371 (e)(3))           <ol style="list-style-type: none"> <li><input type="checkbox"/> are transmitted herewith (required only if not transmitted by the International Bureau).</li> <li><input type="checkbox"/> have been transmitted by the International Bureau.</li> <li><input type="checkbox"/> have not been made; however, the time limit for making such amendments has NOT expired.</li> <li><input checked="" type="checkbox"/> have not been made and will not be made.</li> </ol> </li> <li><input type="checkbox"/> A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).</li> <li><input type="checkbox"/> An oath or declaration of the inventor(s) (35 U.S.C. 371 (c)(4)).</li> <li><input type="checkbox"/> A copy of the International Preliminary Examination Report (PCT/IPEA/409).</li> <li><input type="checkbox"/> A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371 (c)(5)).</li> </ol>					
Items 13 to 18 below concern document(s) or information included:					
<ol style="list-style-type: none"> <li><input type="checkbox"/> An Information Disclosure Statement under 37 CFR 1.97 and 1.98.</li> <li><input type="checkbox"/> An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.</li> <li><input checked="" type="checkbox"/> A <b>FIRST</b> preliminary amendment.           <ol style="list-style-type: none"> <li><b>A SECOND</b> or <b>SUBSEQUENT</b> preliminary amendment.</li> </ol> </li> <li><input type="checkbox"/> A substitute specification.</li> <li><input type="checkbox"/> A change of power of attorney and/or address letter.</li> <li><input type="checkbox"/> Certificate of Mailing by Express Mail</li> <li><input type="checkbox"/> Other items or information:</li> </ol>					

U.S. APPLICATION NO. (IF KNOWN, SEE 37 CFR  
09/341543INTERNATIONAL APPLICATION NO.  
PCT/FR98/02409ATTORNEY'S DOCKET NUMBER  
ATOCM 151

20. The following fees are submitted.

## BASIC NATIONAL FEE (37 CFR 1.492 (a) (1) - (5)) :

<input checked="" type="checkbox"/> Search Report has been prepared by the EPO or JPO .....	\$840.00
<input type="checkbox"/> International preliminary examination fee paid to USPTO (37 CFR 1.482) .....	\$670.00
<input type="checkbox"/> No international preliminary examination fee paid to USPTO (37 CFR 1.482) but international search fee paid to USPTO (37 CFR 1.445(a)(2)) .....	\$760.00
<input type="checkbox"/> Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO .....	\$970.00
<input type="checkbox"/> International preliminary examination fee paid to USPTO (37 CFR 1.482) and all claims satisfied provisions of PCT Article 33(2)-(4) .....	\$96.00

CALCULATIONS PTO USE ONLY

## ENTER APPROPRIATE BASIC FEE AMOUNT =

\$840.00

Surcharge of \$130.00 for furnishing the oath or declaration later than  
months from the earliest claimed priority date (37 CFR 1.492 (e)).

\$130.00

CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE	
Total claims	5 - 20 =	0	x \$18.00	\$0.00
Independent claims	1 - 3 =	0	x \$78.00	\$0.00
Multiple Dependent Claims (check if applicable).				\$0.00
<b>TOTAL OF ABOVE CALCULATIONS</b>				<b>\$970.00</b>
Reduction of 1/2 for filing by small entity, if applicable. Verified Small Entity Statement must also be filed (Note 37 CFR 1.9, 1.27, 1.28) (check if applicable).				<input type="checkbox"/> \$0.00
				<b>SUBTOTAL</b> = <b>\$970.00</b>
Processing fee of \$130.00 for furnishing the English translation later than months from the earliest claimed priority date (37 CFR 1.492 (f)).				<input type="checkbox"/> 20 <input type="checkbox"/> 30 + \$0.00
				<b>TOTAL NATIONAL FEE</b> = <b>\$970.00</b>
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31) (check if applicable).				<input type="checkbox"/> \$0.00
				<b>TOTAL FEES ENCLOSED</b> = <b>\$970.00</b>
				Amount to be: refunded \$ charged \$

 A check in the amount of **\$970.00** to cover the above fees is enclosed. Please charge my Deposit Account No. in the amount of to cover the above fees.  
A duplicate copy of this sheet is enclosed. The Commissioner is hereby authorized to charge any fees which may be required, or credit any overpayment  
to Deposit Account No. **13-3402** A duplicate copy of this sheet is enclosed.

NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.

SEND ALL CORRESPONDENCE TO:

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*I. William Millen*  
SIGNATURE *July 9, 1999*  
30-5-95

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NAME

19,544

REGISTRATION NUMBER

13 July 1999

DATE

09/341543  
80 Rec'd PCT/PTO 13 JUL 1999

**IN THE UNITED STATES DESIGNATED/ELECTED OFFICE**

International Application No. : PCT/FR98/02409 *με*  
International Filing Date : 12 November 1998  
Priority Date Claimed : 14 November 1997  
Applicant(s) (DO/EO/US) : RADIGON, Eric et al.  
Title: PRESSURE-SENSITIVE ADHESIVE WHICH CAN BE APPLIED WHILE HOT

**PRELIMINARY AMENDMENT**

BOX PCT

Assistant Commissioner for Patents  
Washington, D.C. 20231

SIR:

Prior to calculating the national fee, and prior to examination in the National Phase of the above-identified International application, please amend as indicated below.

**IN THE CLAIMS:**

Please **amend claims 4 and 5** as follows:

**Claim 4, line 1 and 2:** Change "any of the preceding claims" to -- Claim 1 --.

**Claim 5, line 1 and 2:** Change "any of the preceding claims" to -- Claim 1 --.

**REMARKS**

The principal purpose of this Preliminary Amendment is to eliminate multiple dependencies in order to avoid extra fees, applicants reserving the right to amend claims to cancelled combined subject.

Respectfully submitted,

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*3c595*

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Filed: 13 July 1999

## CLAIMS

1. Pressure-sensitive adhesive comprising:
  - an ethylene/alkyl (meth)acrylate copolymer (A), the alkyl group having at least 5 carbon atoms,
    - 5 - at least one tackifying resin and optionally a plasticizer,this adhesive being deposited while hot on a substrate.
2. Adhesive according to Claim 1, in which the alkyl group of the alkyl (meth)acrylate has from 6 to 24 carbon atoms.
3. Adhesive according to Claim 1, in which (A) is a copolymer of ethylene and of 2-ethylhexyl acrylate.
4. Adhesive according to any one of the preceding claims, in which the amount of tackifying resin is from 50 to 180 parts (by weight), preferably 100 to 150, per 100 parts of (A).
5. Adhesive according to any one of the preceding claims, in which the amount of plasticizer is 0 from 10 to 30 parts per 100 parts of (A).

ABSTRACT

The present invention relates to a pressure-sensitive adhesive comprising:

- an ethylene/alkyl (meth)acrylate copolymer (A),

- at least one tackifying resin and optionally a plasticizer,  
this adhesive being deposited while hot on a substrate.

These products are useful for self-adhesive labels and tapes.

AM 1273

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**PRESSURE-SENSITIVE ADHESIVE WHICH CAN BE APPLIED  
WHILE HOT**

The present invention relates to a pressure-sensitive adhesive which can be applied while hot.

5 Hot-melt adhesives are thermoplastic materials which are solid at room temperature and which, on heating, become viscous liquids. These viscous liquids are applied to a first substrate and then the substrate is covered with a second surface. On 10 cooling, adhesion is obtained between the substrate and the second surface. The assembly time is the period during which the adhesive which has been applied to a substrate, which is at room temperature, remains tacky, that is to say the interval of time during which it is 15 possible to apply the second surface and, on cooling, to obtain adhesion between the substrate and the second surface.

Once this assembly time period has been exceeded, it is no longer possible to obtain sufficient 20 adhesion between the substrate and the second surface.

These adhesives are denoted by the abbreviation HMA (hot-melt adhesives). Adhesives having an infinite assembly time are useful for self-adhesive labels or adhesive tapes which are used at room 25 temperature. Depending on the nature of the adhesive, it is possible to obtain more or less strong adhesions, for example to detach and reattach the label. Bonding

is achieved by pressure at room temperature. The adhesive is deposited on the substrate (for example, label or tape) while hot. These adhesives are denoted by HMPSA (hot-melt pressure-sensitive adhesives). The 5 present invention relates to this type of adhesive. They are also sometimes denoted as self-adhesive hot-melt compositions.

According to the prior art, see, for example, Ullmann's Encyclopedia of Industrial Chemistry, 5th 10 edition (1995), Vol. A 26, p. 659-660, HMPSAs are preferably based on SIS (styrene/isoprene/styrene) or SBS (styrene/butadiene/styrene) block copolymers.

Mention has been made, in Patent US 5,373,041, relating to HMPSAs, as disadvantages of SBSs 15 and SISs, of their instability towards heat and towards U.V. radiation, as well as of their poor resistance to oils. Provision was thus made, in this patent, to replace SBSs and SISs with copolymers composed of 60 to 70% (by weight) of ethylene, 30 to 40% of n-butyl 20 acrylate and from 0 to 5% of (meth)acrylic acid, which can be neutralized by metal ions.

It has now been discovered that ethylene/alkyl meth(acrylate) copolymers, the alkyl group having at least 5 carbon atoms and advantageously 25 6 to 24 carbon atoms, make it possible to prepare HMPSA adhesives with properties essentially similar to those based on SIS or SBS, while having a better loop tack.

The present invention is thus a pressure-

sensitive adhesive comprising:

- an ethylene/alkyl (meth)acrylate copolymer (A), the alkyl group having at least 5 carbon atoms,
- a tackifying resin and optionally a

5 plasticizer.

These adhesives are deposited while hot (molten) on a substrate, such as, for example, a tape made of paper or made of polyamide, polyolefin or polyester; on cooling, a face is obtained which is 10 adhesive at room temperature. This adhesive face can be protected before use by a silicone-containing paper or the other face of the substrate (rolling up of an adhesive tape on itself).

In the continuation of the text, reference is 15 made to various tests or methods for measuring the properties of HMPASAs:

The loop tack test is the FINAT FTM 9 test described in the FINAT technical manual, Laan Copes Van Cattenbubch 79, NL 2585 EW, THE HAGUE (1995).

20 This test characterizes the instantaneous adhesion or "tack". The tack is defined as being the force required to detach an HMPSA-coated PET (polyethylene terephthalate) loop, a predetermined surface of which has been brought into contact 25 beforehand with a stainless steel plate.

The HMPSA is coated, between 140°C and 180°C, on a 25 x 400 mm<sup>2</sup> PET strip. The thickness being constant, the weight per unit area is between 18 and

22 g/m<sup>2</sup>. The test strips must be conditioned for at least 4 hours before the test in a controlled-environment room at 23 ± 2°C and 50 ± 5% RH (relative humidity). The self-adhesive tape is applied using a 5 standardized 2 kg conventional roller.

The test is carried out using a dynamometer, at a rate of 300 mm/min, in a controlled-environment room at 23 ± 2°C and 50 ± 5% RH.

10 The loop tack is quantified by the value of the maximum force. The result of the creep test is given in N/cm. The type of failure must be mentioned.

The various types of failure are defined as follows:

***Adhesive failure***

15 The HMPSA does not adhere to one of the two substrates.

***Cohesive failure***

Failure in the adhesive seal is observed. In this case, the two adhesively-bonded substrates carry 20 away part of the seal during traction.

***Mixed failure***

Failure is indecisive and both types of failures described above can be observed on the same test specimen.

25 The creep behaviour is determined by the FTM8 test (FINAT manual already mentioned).

The creep test measures the ability of an HMPSA to withstand a static force of 1 kgf at a given

temperature.

The resistance to static shearing is defined by the time necessary to separate, by parallel vertical slippage, an area of  $25 \times 25 \text{ mm}^2$ , coated with HMPSA, 5 from a flat stainless steel plate.

The HMPSA is coated, between  $140^\circ\text{C}$  and  $180^\circ\text{C}$ , on a  $25 \times 400 \text{ mm}^2$  PET strip. The thickness being constant, the weight per unit area is between 18 and 10 22  $\text{g/m}^2$ . The test strips must be conditioned for at least 4 hours before the test in a controlled-environment room at  $23 \pm 2^\circ\text{C}$  and  $50 \pm 5\%$  RH. The self-adhesive tape is applied using a standardized 2 kg conventional roller.

15 The result of the creep test is given in minutes. The type of failure must be mentioned.

Adhesion to steel is determined by the FTM1 test (FINAT manual already mentioned).

20 This test quantifies the adhesiveness. The latter is defined as being the force required to remove a PET strip, coated with HMPSA, from a stainless steel plate.

The HMPSA is coated, between  $140^\circ\text{C}$  and  $180^\circ\text{C}$ , on a  $25 \times 400 \text{ mm}$  PET strip. The thickness being constant, the weight per unit area is between 18 and 25 22  $\text{g/m}^2$ . The test strips must be conditioned for at least 4 hours before the test in a controlled-environment room at  $23 \pm 2^\circ\text{C}$  and  $50 \pm 5\%$  RH. The self-adhesive tape is applied using a standardized 2 kg

conventional roller.

The adhesive force is measured 20 minutes after application. The test is carried out using a dynamometer, at an angle of 180°, at a rate of 5 300 mm/min in a controlled-environment room at 23 ± 2°C and 50 ± 5% RH.

The result of the peel test is given in N/cm. The type of failure must be mentioned.

Cloud point:

10 The cloud point is determined as follows: the adhesive is heated to 175°C and a drop of it is deposited on the bulb of an ASTM thermometer, and then the temperature at which cloudiness appears during cooling is recorded. A value of less than 50°C or 60°C 15 indicates good compatibility between the constituents of the hot melt.

Brookfield viscosity:

Measured at 170°C, needle 27 at 10 revolutions/minute according to ASTM D 3236.

20 SAFT (shear adhesion failure temperature)

The SAFT test (ASTM D 4498) measures the ability of an HMPSA to withstand a static force of 0.5 kg (or 0.25 kg) under the effect of a steady rise in temperature of 0.4°C/min.

25 The SAFT is defined by the temperature at which separation, by parallel vertical slippage, may be observed of an area of 25 × 25 mm<sup>2</sup>, coated with HMPSA, from a flat stainless steel plate.

The HMPSA is coated, between 140°C and 180°C, on a 25 × 400 mm<sup>2</sup> PET strip. The thickness being constant, the weight per unit area is between 18 and 22 g/m<sup>2</sup>. The test strips must be conditioned for at least 4 hours before the test in a controlled-environment room at 23 ± 2°C and 50 ± 5% RH. The self-adhesive tape is applied using a standardized 2 kg conventional roller.

The result of the SAFT is given in °C. The  
10 type of failure must be mentioned.

### Finger tack (internal method)

This test gives an idea of the immediate adhesion of an HMPSA coated onto a PET substrate. The finger tack value is between 0 and 3.

15            0 : no tack,  
              1 : insufficient tack,  
              2 : good tack,  
              3 : excellent tack.

Rolling ball tack test ASTM D 3121 of 1989

20 During the determination of the tack of a dispersion according to the rolling ball tack method, a steel ball is released at the top of an inclined plane.

The ball accelerates and rolls along a horizontal surface covered with the test product. The tack is determined by measuring the distance travelled by the ball before it comes to a halt.

## Equipment

- Device equipped with an inclined plane of

20.0 ± 0.2°

- Steel ball with a mass of 5.60 ± 0.05 g
- Polyethylene terephthalate (PET) strips coated with test products.

5 The ethylene/alkyl (meth)acrylate copolymer (A) is such that the alkyl group contains at least 5 carbon atoms.

10 This is because the Applicant Company has discovered that alkyls having at least 5 carbon atoms give tack to the HMPSA.

The alkyl can be linear, branched or cyclic. It advantageously comprises 6 to 24 carbon atoms. Mention may be made, as example of these alkyl (meth)acrylates, of 2-ethylhexyl (meth)acrylate.

15 The (meth)acrylate content is advantageously at most 50% and preferably between 20 and 40% by weight. The melt flow index (or MFI) can be between 0.1 and 1000 and is preferably at least 200 (in g/10 min according to ASTM D 1238-73, conditions E).

20 It would not be departing from the scope of the invention to use a mixture of at least two ethylene/alkyl (meth)acrylate copolymers.

25 According to another form of the invention, (A) can be a mixture of two copolymers (A<sub>1</sub>) and (A<sub>2</sub>) with different MFI values.

Advantageously, (A<sub>1</sub>) has an MFI of less than 10 and (A<sub>2</sub>) has an MFI of greater than 10. For example, the MFI of (A<sub>1</sub>) is between 1 and 3 and the MFI of (A<sub>2</sub>)

is between 50 and 400.

The adhesives of the invention contain one or more tackifying resins and optionally plasticizers, fillers and stabilizers.

5        Tackifying resins which are suitable are, for example, rosin, rosin esters, hydrogenated rosin, polyterpenes and derivatives, aromatic or aliphatic petroleum resins, or hydrogenated cyclic resins. These resins typically have a ring-and-ball softening 10      temperature of between 25°C and 180°C and preferably between 50°C and 135°C.

The amount of tackifying resin can be from 50 to 180 parts per 100 parts of (A) and preferably 100 to 150 parts.

15        Other examples of rosin derivatives are described in Ullmann's (cited above), Vol. A 23, p. 79-86, the contents being incorporated in the present application.

20        Mention may be made, as derivatives of rosin, of those obtained by hydrogenation, dehydrogenation, polymerization or esterification. These derivatives can be used as is or in the form of esters of polyols, such as esters of pentaerythritol, polyethylene glycol and glycerol.

25        Mention may also be made, as tackifying resin, of dicyclopentadienes.

The plasticizers which can be used in the adhesives of the invention are, for example,

paraffinic, aromatic or naphthenic mineral oils. They serve essentially to lower the viscosity and to introduce tack. The amount of plasticizer can be between 10 and 30 parts per 100 parts of (A).

5        Mention may also be made, as plasticizer, of phthalates, azelates, adipates, tricresyl phosphate and polyesters.

Mention may be made, as examples of fillers, of silica, alumina, glass, glass beads, calcium 10 carbonates, fibres and metal hydroxides. These fillers must not reduce either the tack or the mechanical properties of the adhesive after it has been applied. The amount of fillers can represent up to 100 parts per 100 parts of (A).

15        It is recommended to add stabilizers, such as antioxidants; the usual antioxidants for thermoplastics can be used.

The hot-melt adhesives of the invention are prepared by mixing in the molten state, at temperatures 20 between 130°C and 200°C, until a homogeneous mixture is obtained. The duration of mixing can be of the order of 30 minutes to 3 hours. The usual devices for thermoplastics, such as extruders, rollers, Banbury or Brabender mixers, or propeller mixers, can be used.

25        Examples

The following products were used:  
SIS: styrene/isoprene/styrene block copolymer containing 15% PS, sold by Shell under the reference

Kraton-D-1161 N.

E/2-EHA/3 to 8: ethylene/2-ethylhexyl acrylate (2-EHA) copolymers with characteristics:

	2-EHA weight %	MFI g/min	M.p. °C	Tg °C measured by D.S.C.
E/2-EHA/3	26	3	89	-46
E/2-EHA/4	20	10	89	-36
E/2-EHA/5	25	45	88	-28
E/2-EHA/7	27	120	85	-39
E/2-EHA/8	37	410	73	-42

Catenex N 956: aliphatic paraffinic oil  
(produced by Shell)

Irganox 1010: phenolic antioxidant (produced by Ciba Speciality Chemicals).

15 The properties of the HMPSAs produced with  
SIS (not according to the invention) and according to  
the invention with E/2-EHA copolymers are shown in the  
following tables. The composition of the HMPSA is  
stated at the head of each table.

20 For example, the E/2-EHA/7 column means that  
this is an HMPSA based on the E/2-EHA/7 copolymer.

Table 1

35% (Copolymer (A) or SIS), 40% Permalyin 5095,

25% Catenex N956 and 0.2% Irganox 1010

	TESTS	UNITS	SIS	E/2-EHA/5	E/2-EHA/7	E/2-EHA/8
5	<b>cloud point</b>	°C	< 50	< 50	< 50	< 50
	<b>Brookfield viscosity</b>					
	170°C	mPa·s	9350	8830	3800	2250
	10 r/min, Nee. 27					
10	<b>S.A.F.T.</b>	°C				
	500 g		<30	<30	<30	<30
	250 g		54.9	53.9	58.5	37.9
	s		0.7	1.4	2.0	1.4
	ASTM D 4498					
15	<b>Static creep</b>	min	42	0	17	0
	1000 g, 23°C		9	0	8	0
	s		CF	(AF, Stainless steel)	CF	CF
	Type of failure					
	FTM8					
20	<b>Peel at 180°</b>	N/cm	4.5	2.5	3.3	0.3
	PET/Stainless steel/23°C		0.5	0.2	0.6	0
	s		CF	(AF, Stainless steel)	CF	CF
	Type of failure					
	FTM1					

AF: Adhesive failure; CF: Cohesive failure; MF: Adhesive mixed failure;

25 (AF): Adhesive failure with slight deposition of material on the plate

Table 2

35% (Copolymer (A) or SIS), 40% Permalyne 5095,

25% Catenex N956 and 0.2% Irganox 1010

	TESTS	UNITS	SIS	E/2-EHA/5	E/2-EHA/7	E/2-EHA/8
5	<b>Rolling ball tack on PET</b>	cm	3 0	17 3	10 1	3 0
	<i>PSTC 6</i>					
	<b>Finger tack</b>	-	3	1	2	2
10	<b>Loop tack</b>					
	PET/Stainless steel/23°C	N/cm	3.7 0.7	3.3 0.7	4.8 0.7	2.3 0.8
	<i>s</i>					
	Type of failure		(AF, Stainless steel)	(AF, Stainless steel)	CF	CF
	FTM9					

AF: Adhesive failure; CF: Cohesive failure; MF: Adhesive mixed failure;

15 (AF): Adhesive failure with slight deposition of material on the plate

'3: Strong tack; 2: Moderate tack; 1: Weak tack; 0: No tack

Table 3

30% (Copolymer (A) or SIS), 40% Permalyne 5095,  
 30% Catenex N956 and 0.2% Irganox 1010

	TESTS	UNITS	SIS	T5/E/2-EHA/3	T5/E/2-EHA/4
5	<b>Cloud point</b>	°C	< 50	< 50	< 50
	<b>Brookfield viscosity</b>				
	170°C 10 r/min, Nee. 27	mPa·s	4850	11900	8450
10	<b>S.A.F.T.</b>	°C	49.0 2.0	51.1 4.2	45.1 2.1
	<b>Peel at 180°</b>				
	Mylar/Stainless steel/23°C s	N/cm	6.1 0.1	1.1 0.2	1.3 0.4
	<b>Type of failure</b>		CF	(AF, Stainless steel)	(AF, Stainless steel)
	<b>FTM1</b>				
20	AF: Adhesive failure; CF: Cohesive failure; MF: Adhesive mixed failure; (AF): Adhesive failure with slight deposition of material on the plate				

Table 4

30% (Copolymer (A) or SIS), 40% Permalyne 5095,  
 30% Catenex N956 and 0.2% Irganox 1010

	TESTS	UNITS	SIS	E/2-EHA/3	E/2-EHA/4
5	<b>Rolling ball tack on PET</b> s PSTC 6	cm	3 0	6 1	4 1
	<b>Finger tack'</b>	-	3	1	1.5
10	<b>Loop tack</b> s Type of failure PTM9	N/cm	4.6 0.8 (AF, Stainless steel)	2.3 0.8 (AF, Stainless steel)	2.5 0.6 (AF, Stainless steel)

AF: Adhesive failure; CF: Cohesive failure; MF: Adhesive mixed failure;  
 (AF): Adhesive failure with slight deposition of material on the plate  
 '3: Strong tack; 2: Moderate tack; 1: Weak tack; 0: No tack

Table 5

(15% Copolymer (A<sub>1</sub>) + 15% Copolymer (A<sub>2</sub>) or 30% SIS),  
 40% Permalyn 5095, 30% Catenex N956 and  
 0.2% Irganox 1010

	TESTS	UNITS	30% SIS	E/2-EHA/3 E/2-EHA/5	E/2-EHA/3 E/2-EHA/7	E/2-EHA/3 E/2-EHA/8
5	<b>Cloud point</b>	°C	< 50	< 50	< 50	< 50
10	<b>Brookfield viscosity</b> 170°C 10 r/min, Nee. 27	mPa·s	4100	7200	4830	4700
10	<b>S.A.F.T.</b> 250 g s ASTM D 4498	°C	50.0 0.9	51.7 1.8	52.7 1.2	47.2 1.2
15	<b>Static creep</b> PET/Stainless steel/23°C 1000 g, s Type of failure FTM8	min	238 24 CF	109 10 CF	59 11 CF	31 6 CF
20	<b>Rolling ball tack</b> on PET s PSTC 6	cm	3 1	>40	>40	8 2
	<b>Finger tack</b>	-	3	2	2	2.5

Table 6

(15% Copolymer (A<sub>1</sub>) + 15% Copolymer (A<sub>2</sub>) or 30% SIS),  
 40% Permalyn 5095, 30% Catenex N956 and 0.2%  
 Irganox 1010

5	TESTS	UNITS	30% SIS	E/2-EHA/3	E/2-EHA/3	E/2-EHA/3
			E/2-EHA/5	E/2-EHA/7	E/2-EHA/8	
<b>Loop tack</b>						
s	PET/Stainless steel/23°C	N/cm	7.1	6.1	5.9	3.2
			0.7	0.4	0.2	0.4
			CF	CF	CF	CF
10	Loop tack	N/cm	5.9	4.8	4.6	2.8
			0.2	0.7	0.6	0.3
			CF	CF	CF	CF

AF: Adhesive failure; CF: Cohesive failure; MF: Adhesive mixed failure;

15 (AF): Adhesive failure with slight deposition of material on the plate

Table 7

(15% Copolymer (A<sub>1</sub>) + 15% Copolymer (A<sub>2</sub>) or 30% SIS),  
 40% Permalyne 5095, 30% Catenex N956 and  
 0.2% Irganox 1010

	TESTS	UNITS	30% SIS	E/2-EHA/3 E/2-EHA/5	E/2-EHA/3 E/2-EHA/7	E/2-EHA/3 E/2-EHA/8
<b>Loop tack</b>						
5	PET/Stainless steel/23°C					
	CF					
	s					
10	MF	N/cm			6.9	7.4
	s				2.0	1.1
	AF, Stainless steel		3.3	1.5		
	s		0.8	0.5		
	FTM9					
<b>Loop tack</b>						
15	PET/Glass/23°C					
	CF			11.2		6.2
	s			0.8		0.3
20	MF	N/cm			6.5	
	s				1.5	
	AF, Glass		3.3	1.7	3.2	
	s		0.2	0.2	0.5	
	FTM9					

AF: Adhesive failure; CF: Cohesive failure; MF: Adhesive mixed failure;

25 (AF): Adhesive failure with slight deposition of material on the plate

CLAIMS

1. Hot-melt adhesive comprising:
  - an ethylene/alkyl (meth)acrylate copolymer5 (A), the alkyl group having at least 5 carbon atoms,
  - at least one tackifying resin, optionally a plasticizer and optionally a wax,this adhesive being deposited while hot on a substrate.
2. Adhesive according to Claim 1, in which 10 the alkyl group of the alkyl (meth)acrylate has from 6 to 24 carbon atoms.
3. Adhesive according to Claim 1, in which (A) is a copolymer of ethylene and of 2-ethylhexyl acrylate.
- 15 4. Adhesive according to any one of the preceding claims, in which the amount of tackifying resin is from 50 to 180 parts (by weight), preferably 100 to 150, per 100 parts of (A).
- 20 5. Self-adhesive hot-melt pressure-sensitive adhesive (HMPSA) according to any one of the preceding claims comprising:
  - the copolymer (A)
  - at least one tackifying resin
  - at least one plasticizer25
  - optionally stabilizers.
6. Hot-melt adhesive (HMA) according to any one of Claims 1 to 4 comprising:

- the copolymer (A)
- at least one tackifying resin
- at least one wax
- optionally stabilizers.

Docket No.  
ATOCM 151

# Declaration and Power of Attorney For Patent Application

## English Language Declaration

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name,

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled

### PRESSURE-SENSITIVE ADHESIVE WHICH CAN BE APPLIED WHILE HOT

the specification of which

(check one)

is attached hereto.

was filed on 12 NOVEMBER 1998 as United States Application No. or PCT International Application Number PCT/FR98/02409  
and was amended on \_\_\_\_\_

(if applicable)

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose to the United States Patent and Trademark Office all information known to me to be material to patentability as defined in Title 37, Code of Federal Regulations, Section 1.56.

I hereby claim foreign priority benefits under Title 35, United States Code, Section 119(a)-(d) or Section 365(b) of any foreign application(s) for patent or inventor's certificate, or Section 365(a) of any PCT International application which designated at least one country other than the United States, listed below and have also identified below, by checking the box, any foreign application for patent or inventor's certificate or PCT International application having a filing date before that of the application on which priority is claimed.

### Prior Foreign Application(s)

Priority Not Claimed

97/14320

FRANCE

14 NOVEMBER 1997

(Number)

(Country)

(Day/Month/Year Filed)

(Number)

(Country)

(Day/Month/Year Filed)

(Number)

(Country)

(Day/Month/Year Filed)

I hereby claim the benefit under 35 U.S.C. Section 119(e) of any United States provisional application(s) listed below:

(Application Serial No.)

(Filing Date)

(Application Serial No.)

(Filing Date)

(Application Serial No.)

(Filing Date)

I hereby claim the benefit under 35 U. S. C. Section 120 of any United States application(s), or Section 365(c) of any PCT International application designating the United States, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT International application in the manner provided by the first paragraph of 35 U.S.C. Section 112, I acknowledge the duty to disclose to the United States Patent and Trademark Office all information known to me to be material to patentability as defined in Title 37, C. F. R., Section 1.56 which became available between the filing date of the prior application and the national or PCT International filing date of this application:

(Application Serial No.)

(Filing Date)

(Status)  
(patented, pending, abandoned)

(Application Serial No.)

(Filing Date)

(Status)  
(patented, pending, abandoned)

(Application Serial No.)

(Filing Date)

(Status)  
(patented, pending, abandoned)

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

POWER OF ATTORNEY: As a named inventor, I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and transact all business in the Patent and Trademark Office connected therewith. (list name and registration number)

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